

## Evaluation of Resin-Matrix Ceramic Bond Durability to Self-adhesive Resin Cement with Different Universal Adhesives

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### Abstract

The study determines the durability of different universal adhesives for surface treatment of resin-matrix ceramic luted to self-adhesive resin cement. Sixty ceramic blocks were produced, sandblasted, and assigned into ten specimens in each group and surface treated with five universal adhesives [Scotchbond universal plus (SUP), Beautibond Xtreme (BXT), Singlebond universal (SBU), Clearfil Tri-S bond universal (CTU), and Tetric N-bond universal (TNU)] and luted with resin cement [RelyX unicem (RXU)] as follows: group 1, RXU; group 2, SUP+RXU; group 3, BXT+RXU; group 4, SBU+RXU; group 5, CTU+RXU; group 6, TNU+RXU. A silicone mold was put on the sample center, then injected with RXU. The samples were aged on thermocycling instrument. A mechanical testing device was used to calculate the samples' micro-shear bond strength (MSBS).

To analyze failure patterns, a stereomicroscope was employed to determine the debonded surfaces. To determine MSBS from the data, a one-way ANOVA was used, and the significant level ( $P < 0.05$ ) was determined with Tukey's test. Groups 2 ( $20.48 \pm 1.57$  MPa) and 3 ( $19.85 \pm 1.95$  MPa) obtained the two highest MSBS, with no significant difference between them. Group 1 was able to obtain the lowest MSBS ( $8.06 \pm 1.36$  MPa). A high MSBS was commonly associated with mixed failures.

The best method to improve the MSBS is to apply universal adhesive before cementation with self-adhesive resin cement. Additionally, the best method to achieve maximum MSBS is to apply universal adhesive containing mixed silane (SUP) and ARS (BXT) prior to cementation with self-adhesive resin cement.

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### Introduction

In the field of restorative dentistry, the need for all-ceramic restorations has increased, leading to a decrease in the use of traditional metallic materials. Because they resemble natural teeth, all-ceramic restorations are very esthetically pleasing and satisfy patient and

dentist needs. Resin-matrix ceramics are becoming increasingly common in dental clinical practice because of their great mechanical qualities, high biocompatibility, and outstanding results.<sup>1,2</sup> This correlates with the growing popularity of computer-aided design/computer-aided manufacturing (CAD/CAM) innovation in prosthodontic and restorative dentistry, where dentists can create the restoration in one appointment. As a result, a wide variety of uses in restorative and prosthodontic dentistry, such as fixed prostheses and single crowns, are made possible by resin-matrix ceramic.<sup>3</sup>

The main phase of the resin-matrix ceramic is inorganic.<sup>4</sup> It is made up of two consecutive interpenetrating connections, one made of ceramic substance and one composed of the polymer, generated by the polymer

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penetrating through a porosity ceramic structure.<sup>5</sup> When two phases of a resin-matrix material have formed, interfacial crack deflection usually limits the propagation of cracks.<sup>5</sup> Currently, the resin-matrix ceramic that is supplied commercially is known as Shofu block HC (Shofu, Kyoto, Japan). Urethane dimethacrylate (UDMA), triethylene glycol dimethacrylate (TEGDMA), zirconium silicate, silica powder, and micro-furmed silica were used to create this material.<sup>6,7</sup>

Numerous surface alterations have been performed to promote the resin cement's bonding capacity to resin-matrix ceramics. The proper surface modification of indirect restoration substances prior to luting cementation is generally acknowledged to be essential for enhancing the luting cements' adherence to restorations and, as a result, extending the restorations' lifespan.<sup>8</sup> Nevertheless, the primary factor influencing the surface modification technique is the material's chemical composition. For the resin-matrix ceramic (Shofu block HC), sandblasting is the technique most frequently employed for micro-mechanical retention.<sup>6,7</sup> The sandblast method of surface modification produces a rougher surface, which strengthens the interaction of the Shofu block HC and the hydrophobic resin substance.<sup>6</sup> The chemical adhesion for Shofu block HC is achieved by the HC primer applied before the adhesive agent; that is the standard protocol for pretreatment of Shofu block HC.<sup>6</sup> The prior investigations found that the pretreatment of Shofu block HC with universal adhesive is the best alternative surface modification method and enhances the bond capacity between resin materials and resin-matrix ceramic.<sup>6</sup> Assessing the adhesive systems' adhering capacity to a resin-matrix ceramic is an approach to determining how successfully a surface treatment is working. Nevertheless, in the oral condition, restorations undergo exposure to thermal, chemical, and mechanical stresses that may degrade the bond quality and the adhesive interaction.<sup>9</sup> Therefore, using the aging process to mimic these settings in in vitro experiments is crucial. While a number of studies have documented various surface treatments performed on resin-matrix ceramics,<sup>6,7,10</sup> the most effective surface modification protocol for Shofu block HC with different universal adhesives and self-adhesive resin cements is still under investigation.

Hence, in the present investigation, we purposely

determine the durability of different universal adhesives for Shofu block HC surface treatment luted to self-adhesive resin cement by assessing their micro-shear bond strengths (MSBS). The null hypothesis states that the different universal adhesives do not significantly affect the MSBS between Shofu block HC and self-adhesive resin cement.

## Materials and methods

### Shofu block HC preparation

This study focused on CAD/CAM hybrid ceramic materials, specifically Shofu Block HC (Shofu, Kyoto, Japan). Sixty specimens (6 × 7 × 1.5 mm) were produced using the preliminary CAD/CAM blocks via a slow-speed diamond cutter using cooling from water (Accuton-50, Struers, Ohio, USA). The samples were set inside an epoxy resin-contained polyvinyl chloride pipe. In order to appropriately modify the roughness of the surface, a sandpaper sheet (3M, MN, USA) was used to polish the specimen surfaces with a silicon carbide grit of 600. After that, the specimens had been ultrasonically treated for ten minutes in distilled water using an ultrasonic cleaner (Eurosonic 4D, Euronda, Italy) to get rid of any surface contamination. Table 1 presents the resin components used in the current investigation.

### Sandblast method

Sixty Shofu block HC specimens were exposed to air abrasion for 10 seconds at 2 bar of pressure using 50-micron alumina oxide (Al<sub>2</sub>O<sub>3</sub>) particles. The air-abrasion device tip was placed 1 cm from the Shofu block HC surface perpendicularly.<sup>11</sup> The materials were sandblasted, washed, and treated with air drying for half a minute via a triple syringe.

### Universal adhesive treatment

The sample's surface was agitated with the universal adhesive using a microbrush for around 20 seconds. Any remaining universal adhesive materials were removed with a clean microbrush. The solvent disappeared after the universal adhesive evaporated via air drying for around five seconds. After that, they were allowed to blow it dry using the air until the universal adhesive stopped flowing and the surface appeared shiny.<sup>6</sup> Then, it obtained a light activation about 20 seconds (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein).

### Dual-cured self-adhesive resin cement application protocol

Random distributions were designed for six groups (n = 10/group) based on the different universal adhesive resin-matrix ceramic surface-treated specimens. The one dual-cured self-adhesive resin cement (RelyX Unicem, RXU) and five universal adhesives [Scotchbond universal plus (SUP), Beautibond Xtreme (BXT), Singlebond universal (SBU), Clearfil Tri-S bond universal (CTU), and Tetric N-bond universal (TNU)] were used in the following ways:

- Group 1: RXU
- Group 2: SUP + RXU
- Group 3: BXT + RXU
- Group 4: SBU + RXU
- Group 5: CTU + RXU
- Group 6: TNU + RXU

The center of the surface-treated specimen was defined by the placement of a 2.0 mm thick and 2.0 mm diameter silicone model. In following the manufacturer's guidelines, the self-adhesive resin cement (RXU) was mixed and injected using a silicone model to the ceramic-treated surface with a force of 50 N using a modified durometer. A new microbrush was employed to remove any residual resin cement, and the resin cement went through polymerization for 40 seconds. Following the removal of a silicon model, 40 seconds of light activation were performed again. Each specimen was incubated in an experimental incubator chamber (Human Lab Inc., Gyeonggi-Do, Korea) in distilled water for one whole day at 37 degrees Celsius.

### Aging protocol

The Shofu block HC-bonded samples were aged on thermocycling equipment (Proto-Tech, Micoforce, OR, USA). Considering 30 seconds of dwell time and 5 seconds of transmission time, the material was thermally cycled 10,000 cycles across 5°C and 55°C.

### Measurement of MSBS and failure pattern investigation

The AGS-X 500N universal measuring instrument (Shimadzu Corporation, Kyoto, Japan) was employed to calculate the MSBS data using a laboratory speed of 0.5 mm/minute, adopting a knife-edge blade (Figure 1). The bonding zone's area and the bond breakage strength were divided in order to calculate the MSBS score.

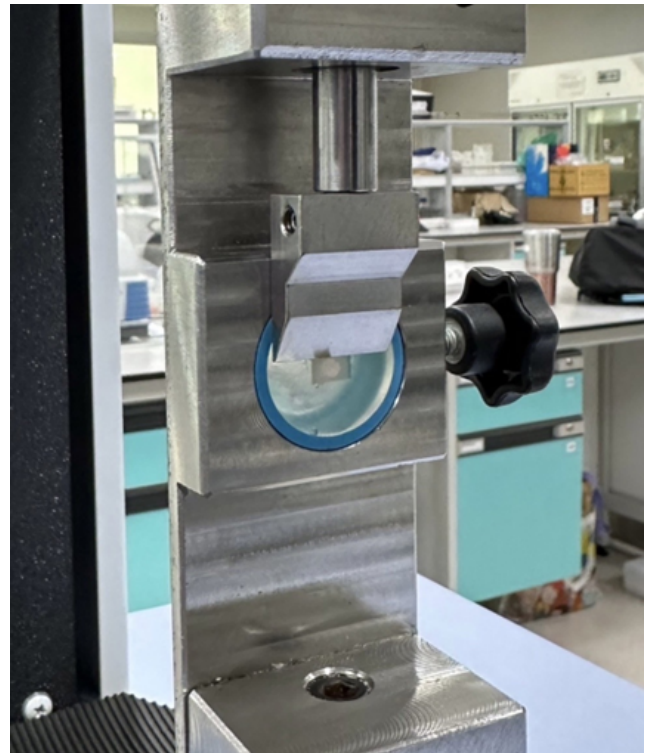


Figure 1. The configuration for the MSBS test.

The Shofu block HC and self-adhesive resin cements' fracture mode profiles were investigated via a stereomicroscope featuring a x40 magnification. Three configurations were developed in order to establish the fracture mechanisms:<sup>12,13</sup> (A) an adhesive pattern (debonded on the junction between Shofu block HC and self-adhesive resin cement), (B) a cohesive pattern (fracture inside Shofu block HC or self-adhesive resin cement), and (C) a mixed pattern (fractures include both Shofu block HC and self-adhesive resin cement).

### The statistical assessment of the data

The normality of the MSBS value pattern was examined by the Kolmogorov-Smirnov test. A one-way analysis of variance (ANOVA) was used to examine each group's MSBS in order to identify any significant variations between the different universal adhesives for Shofu block HC surface treatment and self-adhesive resin cement. To assess MSBS data in MPa, Tukey HSD comparison tests were employed. A significance level of 0.05 and a 95% confidence level were taken into account for all analyses.

### Results

The mean and standard deviations (SD) of MSBS data (MPa) are reported in Table 2. The

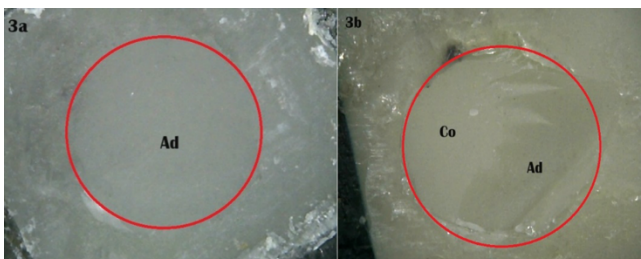
two greatest MSBS values were achieved by groups 2 ( $20.48 \pm 1.57$  MPa) and 3 ( $19.85 \pm 1.95$  MPa), with no significant difference between them. Group 1 succeeded in finding a considerably minimal MSBS value ( $8.06 \pm 1.36$  MPa). Group 5 ( $13.79 \pm 1.71$  MPa) and group 6 ( $13.43 \pm 1.40$  MPa) did not exhibit a statistically significant difference from group 4 ( $14.22 \pm 0.86$  MPa).

Table 2 contains a succinct description of the failure-type frequency structure. Following their breakage, every sample in group 1 that had fractures had an adhesive failure pattern. Furthermore, groups 2 through 6 addressed mixed failure situations. With 60% of the total, groups 2 and 3 showed the largest percentage of mixed failure structures.

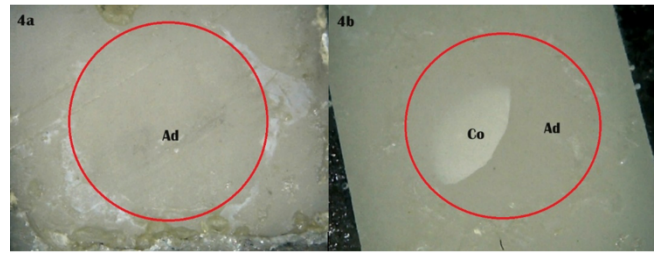
In this part of the work, stereomicroscope pictures are used for clarifying the configurations of adhesive and mixed failure modes. The pictures captured by the stereomicroscope are shown in Figures 2 to 7.



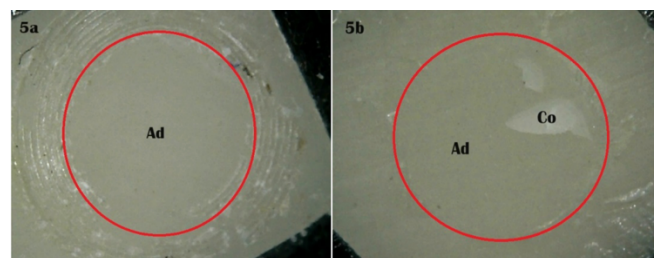
**Figure 2.** Failure mode of group 1 (Ad, adhesive failure).



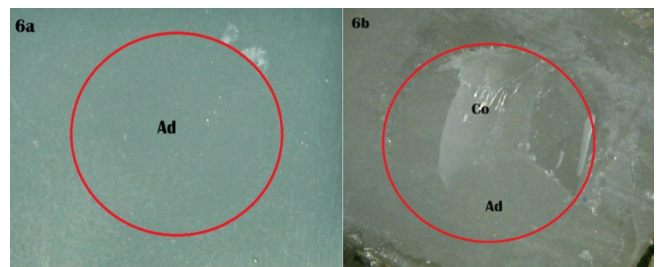
**Figure 3.** Failure mode of group 2; 3a, adhesive failure mode; 3b, mixed failure mode (Ad, adhesive failure; Co, cohesive failure in resin cement).



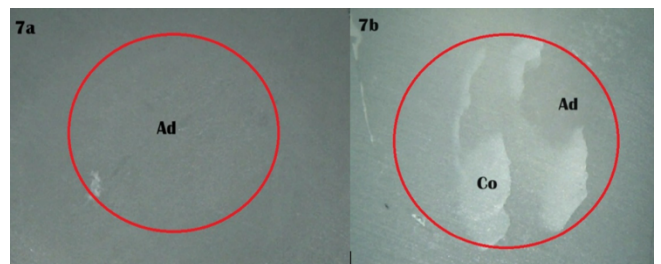
**Figure 4.** Failure mode of group 3; 4a, adhesive failure mode; 4b, mixed failure mode (Ad, adhesive failure; Co, cohesive failure in resin cement).



**Figure 5.** Failure mode of group 4; 5a, adhesive failure mode; 5b, mixed failure mode (Ad, adhesive failure; Co, cohesive failure in resin cement).



**Figure 6.** Failure mode of group 5; 6a, adhesive failure mode; 6b, mixed failure mode (Ad, adhesive failure; Co, cohesive failure in resin cement).



**Figure 7.** Failure mode of group 6; 7a, adhesive failure mode; 7b, mixed failure mode (Ad, adhesive failure; Co, cohesive failure in resin cement).

#### Discussion

The present analysis determines the durability of different universal adhesives for surface treatment of resin-matrix ceramic (Shofu block HC) luted to self-adhesive resin cement by assessing their MSBS. The results indicated significant discrepancies in the MSBS scores for each of the groups. Thus, the null hypothesis had been proven to be incorrect.

The aging of the interactions between resin materials and resin-matrix ceramic is an essential issue to take into account. Due to water's small particle size and high concentration at the molar level, which may pass through functional groups or tiny gaps in polymer chains to reduce the polymer's thermal stability and cause plasticization,<sup>14</sup> the storage in water can have a direct effect on the duration that restorations remain. All of the surface treatments' bond strength values might be lowered by the aging process.<sup>12</sup> Several thermocycling regimens have been used to stimulate the aging process; nevertheless, Gale et al. reported that a thermal cycling approach of 10,000 times annually would be adequate for demonstrating bonding interface failure of the restoration.<sup>15</sup>

Adhesion, as it is currently understood, often hinges on the relationship between micro-mechanical bonding and chemical bonding. To determine a successful relationship between resin materials and resin-matrix ceramic, it is important to comprehend how varied surface modifications affect their interaction. Regarding the Shofu block HC ceramic, a previous report found that the sandblasting method improved the MSBS as compared to hydrofluoric (HF) etching when using universal adhesive, and it is the most efficient way of achieving micro-mechanical bonding.<sup>6,7</sup> The reason is that sandblasting significantly raises the roughness and surface energy of the Shofu block HC. As an alternative, sandblasting elevates the bonding ability through the exposure of inorganic filler parts within the resin matrix. This promotes the creation of a siloxane adhesion bond between the silanol of the silane coupling agent and the inorganic parts of ceramic.<sup>16-18</sup> According to the chemical surface alterations, the HC primer is designed to be used to promote chemical bonding to shofu block HC CAD/CAM restorations to provide long-lasting adhesive bonding.<sup>6,19,20</sup> However, previous studies have shown that the most effective alternative surface alteration technique for Shofu

block HC is to pretreat it with universal adhesive, which promotes the bonding interaction between resin materials and resin-matrix ceramic, which is no significant difference when compared to treated with HC primer.<sup>6</sup> The phosphate functional monomer may chemically attach to the zirconium filler in Shofu block HC because it may form a direct chemical connection with the zirconium oxide.<sup>12</sup> Due to these factors, there was not a significant difference in the HC primer-treated group's binding performance compared to the universal adhesive-treated group. The current examination employs the sandblast method for micro-mechanical bonding and universal adhesives for chemical surface alteration of Shofu block HC, in accordance with the findings of prior research.<sup>6,7</sup>

Our finding indicates that the resin-matrix ceramic (Shofu block HC) surface bonded with dual-cured self-adhesive resin cement, RelyX unicem (RXU), not surface modification with universal adhesive, demonstrated the significantly lowest MSBS ( $8.06 \pm 1.36$  MPa) when compared to the other group. RXU, contained in a phosphate functional monomer, can cause chemical adhesion to the zirconium particle in Shofu block HC. But because it is a high-viscosity material,<sup>21</sup> it cannot penetrate into the sandblasted ceramic surface, giving it the lowest bonding ability. Moreover, the application of the universal adhesive before cementation with the self-adhesive resin cement has a significantly higher MSBS compared to not applying the universal adhesive. Because of its low viscosity, universal adhesive allows it to easily infiltrate into the sandblasted material, which creates strong bonding efficiency.

For the universal adhesive treatment groups, SBU ( $14.22 \pm 0.86$  MPa), CTU ( $13.79 \pm 1.71$  MPa), and TNU ( $13.43 \pm 1.40$  MPa) have a significantly lower MSBS compared to SUP ( $20.48 \pm 1.57$  MPa) and BXT ( $19.85 \pm 1.95$  MPa). The SBU and CTU are composed of a 3-methacryloxypropyltrimethoxysilane (3-MPS) silane agent and a 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) monomer, but the 3-MPS is inefficient. According to several studies,<sup>6,22,23</sup> universal adhesives incorporating silane with mildly acidic pH values might have poor bonding performance as a result of the silane agents' cyclic self-condensation process, based on the form of silane that the universal adhesive contains. There are just 10-MDP

monomers in TNU. Because 10-MPD is made up of a phosphate bifunctional molecule that has the ability to chemically adhere to zirconium oxide,<sup>12</sup> it may chemically attach to the zirconium particle of Shofu block HC. Regarding these explanations, the SBU and CTU bond abilities did not differ significantly from the TNU test. The SUP consists of mixed silane (3-methacryloxypropyltriethoxysilane (3-MPTES) and 3-(aminopropyl)triethoxysilane (APTES)) and 10-MDP. The BXT consists of an acid-resistant silane coupling agent (ARS) and carboxylate and phosphate monomers. Many findings<sup>6,23,24</sup> indicate that the universal adhesive containing mixed silane (3-MPTES and APTES) or ARS has the most effective adhesion performance with a mildly acidic pH value because it protects the silane agent from cyclic self-condensation. The elevation in the MSBS of SUP and BXT might result from the silane agent adhering to the exposed SiO<sub>2</sub> particles in Shofu block HC.<sup>6</sup> The four methods listed below have the potential to promote the bond capacity between Shofu block HC treated with universal adhesive (SUP, BXT)

and self-adhesive resin cement: (i) the ceramic surface that has been sandblasted can be penetrated by the universal adhesive due to its low viscosity. (ii) the 10-MDP molecule of the universal adhesive may chemically stimulate bonding to the zirconium particle in the Shofu block HC. (iii) the silane agent adhering to the exposed SiO<sub>2</sub> particles in Shofu block HC, and (iv) copolymerization of the resin monomers in the universal adhesive with the self-adhesive resin cement is a potential outcome.<sup>25,26</sup>

The results of the MSBS testing agreed with the conclusions established by the analysis of the breakdown mechanisms of the debonded samples. In this investigation, adhesive breaks occurred more often in every group. Groups 2 through 6 that were treated with universal adhesive indicated mixed breakdowns and had stronger MSBS values. Moreover, the SUP and BXT groups had the highest mixed failure rates. Adhesive failure is often the source of low bond ability, while mixed failure indicates stronger adhesion.<sup>6,27-30</sup>

Material	Manufacturer	Chemical composition
Shofu Block HC	Shofu, Kyoto, Japan	UDMA, TEGDMA, Filler; Silica powder, micro fumed silica, zirconium silicate, 61% by weight
RelyX Unicem	3M ESPE, MN, USA	Phosphoric acid methacrylates, dimethacrylates, silanated fillers, inorganic fillers (72 wt.%), initiators, stabilizers, rheologic additives
Scotchbond universal plus	3M, Neuss, Germany	HEMA, 2-propenoic acid, 2-methyl-, diesters with 4,6-dibromo-1,3-benzenediol 2-(2-hydroxyethoxy)ethyl 3-hydroxypropyl diethers, 2-propenoic acid, 2-methyl-, reaction products with 1,10-decanediol and phosphorus oxide, 2-propenoic acid, 2-methyl-, 3(triethoxysilyl)propyl ester, reaction products with silica and 3(triethoxysilyl)-1-propanamine, synthetic amorphous silica, fumed, crystalline-free, ethanol, water, (3-aminopropyl)triethoxysilane, camphorquinone, N,N-dimethylbenzocaine, methacrylic acid, Acetic acid, copper(2+) salt, monohydrate
Beautibond Xtreme	Shofu, Kyoto, Japan	Phosphate ester, dithiooctanoate and carboxylic acid monomers, acid resistant silane coupling agent, acetone
Singlebond universal	3M, Neuss, Germany	10-MDP, Bis-GMA, HEMA, DMA, methacrylate functional copolymer, silane, filler, initiators, ethanol, water
Clearfil Tri-S bond universal	Kuraray Noritake Dental Inc., Okayama, Japan	10-MDP, HEMA, bisphenol A diglycidylmethacrylate, ethanol, hydrophilic amide monomers, colloidal silica, silane coupling agent, sodium fluoride, di-Camphorquinone, water
Tetric N-bond universal	Ivoclar Vivadent, Schaan, Liechtenstein	10-MDP, Bis-GMA, HEMA, UDMA, ethanol, diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide

**Table 1.** presents the resin components used in the current investigation.

**Abbreviations:** UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA, bisphenol A-glycidyl methacrylate; DMA, dimethacrylate.

Groups	Mean MSBS ± SD	Failure pattern		
		Adhesive	Mixed	Cohesive
1. RXU	8.06 ± 1.36 <sup>a</sup>	100	0	0
2. SUP + RXU	20.48 ± 1.57 <sup>b</sup>	50	50	0
3. BXT + RXU	19.85 ± 1.95 <sup>b</sup>	50	50	0
4. SBU + RXU	14.22 ± 0.86 <sup>c</sup>	70	30	0
5. CTU + RXU	13.79 ± 1.71 <sup>c</sup>	70	30	0
6. TNU + RXU	13.43 ± 1.40 <sup>c</sup>	60	40	0

**Table 2.** The mean MSBS ± SD and failure pattern percentage.

A value with the same letters is not different statistically significantly.

## Conclusions

The current in vitro study's findings suggest that the best approach to improving the MSBS is to apply universal adhesive before cementation with self-adhesive resin cement. Furthermore, the best method to achieve maximum MSBS is to apply universal adhesive containing mixed silane (SUP) and universal adhesive containing ARS (BXT) prior to cementation using self-adhesive resin cement.

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## Declaration of Interest

The authors report no conflict of interest.

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